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Effect of Citrate and Carbonate Based Detergents on Wastewater Characteristics and Treatment

Research Report No. 61



**Research Program for the Abatement of Municipal Pollution
under Provisions of the Canada-Ontario Agreement
on Great Lakes Water Quality**

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EFFECT OF CITRATE AND CARBONATE BASED DETERGENTS
ON WASTEWATER CHARACTERISTICS AND TREATMENT

by

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RESEARCH PROGRAM FOR THE ABATEMENT
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ABSTRACT

This study examined the effects of citrate and carbonate based detergents on primary and activated sludge waste treatment processes when phosphorus removal by chemical precipitation was practised. Jar tests, continuous flow bench scale activated sludge reactors, as well as pilot scale and full scale treatment systems, were used in this investigation.

Results showed that for primary treatment systems a complete citrate substitution may increase chemical (ferric chloride, alum or lime) requirements for phosphorus removal, whereas a complete carbonate substitution would have no significant effect on systems using lime for phosphorus removal.

In general, for activated sludge systems the impact upon phosphorus removal and treatment plant performance by a complete substitution to citrate based detergents was discerned to be negligible.

RÉSUMÉ

Le présent rapport étudie les effets des détergents à base de citrates et de carbonates sur le traitement soit primaire, soit par boues activées, des eaux usées qu'on veut déphosphater par précipitation chimique. On a fait des épreuves statiques en récipients, des essais dynamiques en laboratoire avec des réacteurs à boues activées, ainsi que des expériences pilotes et à l'échelle réelle.

Les résultats montrent que, pour le traitement primaire, la présence de détergents aux citrates, uniquement, peut augmenter la consommation de produits chimiques (chlorure ferrique, alun ou chaux) pour la déphosphatation; celle de détergents aux carbonates, uniquement, n'aurait aucun effet important sur le procédé de déphosphatation à la chaux.

En général, pour ce qui est du traitement aux boues activées, la présence de détergents à base de citrates, uniquement, n'a que des effets négligeables sur la déphosphatation et le rendement de l'usine.

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CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

Phosphorus-free detergents, one using citric acid (20% by weight) as builder and one using sodium carbonate (71% Na_2CO_3 by weight) as builder, were evaluated for their effects on sewage treatment processes. The major conclusions from this study were:

1. Jar test results showed that the full scale use of a citrate based detergent may increase the chemical requirements for phosphorus removal in primary treatment systems. This interference with phosphorus removal was shown to be caused by complexing of the precipitant cation (i.e., Fe^{3+} , Al^{3+} , Ca^{2+}) at citric acid concentrations above 10 mg/l.
2. Jar test and pilot scale results showed that if a carbonate based detergent were to be substituted for all detergents now in use, the impact on primary treatment systems using lime for phosphorus removal would be insignificant.
3. Bench scale and full scale activated sludge studies using citrate based detergent showed some reduction in citrate degradation at temperatures less than 10°C. However, citric acid removals still averaged 90% in the activated sludge plant during fall and winter operation.
4. No evidence of increased heavy metal (Cu^{2+} , Zn^{2+} , Ni^{2+}) transport through the activated sludge system, due to citrate addition, could be found in either the bench scale or full scale studies.
5. Results of the full scale citrate addition study indicated no increased precipitant chemical requirements for phosphorus removal in an activated sludge plant. The different effects on primary and secondary systems was to be expected because of the high citrate removal by activated sludge treatment.

6. In general, for biological systems the impact on phosphorus removal and treatment plant performance by a complete substitution to citrate based detergents was discerned to be negligible.
7. A survey of 32 Ontario treatment plants showed a mean influent baseline citrate level of 1.1 mg/l. Primary and secondary treatment reduced this by an average of 55% and 92% respectively.

RECOMMENDATIONS

Citric acid and sodium carbonate based detergents used in these studies had a minimal impact on the operation of wastewater treatment facilities. Based on these findings; the fact that citric acid is readily degradable and that carbonate is normally present in abundance in natural waters, the use of these materials as a substitute for phosphorus builders in detergents could be considered. Other health and environmental considerations, however, may be expected to affect any decision to utilize these materials on such a massive scale as in detergents.

The effect of citric acid on phosphorus removal in a full scale primary treatment facility should be investigated since jar testing has shown that some complexing of the phosphorus precipitant chemical and thus interference with phosphorus removal may occur.

Phosphorus has been identified as a limiting nutrient for algal growth in many lakes and rivers. The removal of phosphorus from wastewater effluents is thus considered essential for reducing the eutrophication potential of these effluents on receiving waters (International Joint Commission, 1970). One approach is to reduce the phosphorus concentrations in detergent formulations. Various studies have shown that phosphorus removal from detergents would decrease by 25% to 50% the total phosphorus loadings received at wastewater treatment plants (e.g., Shannon and Kamp, 1973). However, the sodium tripolyphosphate builder in the detergent formulation must be replaced by some other more innocuous sequestering agent. Some of the more frequently proposed builders are citric acid (H_3Cit), sodium carbonate and nitrilotriacetic acid (NTA).

In recent years, NTA has been studied extensively and is presently in use in Canada and other countries as a replacement for sodium tripolyphosphate. Studies at the Wastewater Technology Centre (Shannon et al, 1974, and Wei et al, 1976) and elsewhere, have indicated that NTA degradation under Canadian winter conditions is reduced to approximately 50%. Furthermore, this relatively poor degradation response may contribute to transport of some heavy metals through sewage treatment facilities (Shannon et al, 1976). The environmental implications of NTA, including possible toxicological effects, have been discussed in detail by Thom (1971), Epstein (1972) and Prakash (1976).

Carbonate builders in detergent formulations increase the alkalinity and pH of wastewaters, but since carbonate is already present in large quantities in natural waters, the overall effect is minimal. While Silberman (1975) found no negative effects of carbonate detergents on biological waste treatment systems, it is postulated that increased carbonate levels may interfere with phosphorus removal systems which utilize lime as a precipitant.

Citric acid has found limited use in European countries in detergent formulations and has been used in some liquid formulations in the United States. Citric acid is considered to be almost completely

biodegradable. Shannon and Kamp (1973) found that in excess of 95% of the citric acid present in citrate based detergent (7.0 mg/l influent) when fed to an activated sludge plant, was removed. No quantifiable effect on phosphorus removal based on jar tests conducted over a short (six-day) period was evident. Citric acid is a moderately strong sequestering agent for metals, and at high concentrations may be expected to solubilize iron and aluminum in a phosphorus removal system. As well, it could assist in transporting other heavy metals through sewage treatment plants. Some preliminary data gathered on citrate metal complex degradation by Phizer Inc., (Foulds, 1973), has indicated that nickel and copper complexes do not degrade readily in activated sludge systems.

The major objective of this study was to evaluate the effect of citric acid and sodium carbonate based detergents on phosphorus removal processes, and to determine their general effects on wastewater treatment plant efficiency.

A secondary objective was to conduct a survey of citrate baseline levels in raw and treated domestic wastewater.

Bench, pilot and full scale studies were conducted. The bench scale studies consisted of jar testing using citric acid and carbonate based detergent additions in order to assess phosphorus removal interference when using ferric chloride, alum or lime. In addition, bench scale continuous flow activated sludge reactors were used to evaluate temperature effects on citrate detergent biodegradability and metal (Cu^{2+} and Ni^{2+}) transport.

Pilot scale evaluation of the carbonate based detergent for phosphorus removal interference using lime was conducted with a 131 m³/d (20 gpm) primary pilot plant.

At full scale, a conventional activated sludge plant was used to evaluate citrate detergent biodegradability, metal (Cu^{2+} and Zn^{2+}) transport, phosphorus removal interference and general treatment process response when using ferric chloride or alum.

An experimental program outline is summarized in Table 1, with details of each experimental phase described in the following subsections.

2.1 Bench Scale Studies - Jar Testing

Jar testing was carried out to determine the effect of the citrate based detergent on phosphorus removal using ferric chloride, aluminum sulphate and lime precipitants. A 25-litre grab sample of degrittied raw sewage was collected from the Burlington Skyway Sewage Treatment Plant and 18 one-litre aliquots taken. Using this approach, four coagulant doses and four detergent concentrations were tested along with two baseline runs. A typical set of detergent and coagulant conditions is shown in Table 2.

The jar test procedure used was as follows:

1. detergent added and mixed;
2. coagulant added and flash mixed for five minutes;
3. flocculated at 40 rpm for 10 minutes;
4. settled at 15 rpm for 90 minutes;
5. quiescent settling for 10 minutes; and
6. samples taken for total phosphorus determinations.

TABLE 1. EXPERIMENTAL PROGRAM OUTLINE

Scale of Investigation	System Type	Detergent Base	
		Citrate	Carbonate
Bench Scale	Jar Tests	• P-removal interference using FeCl_3, $\text{Ca}(\text{OH})_2$ $\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$	• P-removal interference using FeCl_3, $\text{Ca}(\text{OH})_2$ $\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$
	Activated Sludge	• temperature effect on detergent biodegradability and metal transport (Cu^{2+}, Ni^{2+})	
Pilot Scale	Primary Treatment		• P-removal interference using low and high lime systems
Full Scale	Activated Sludge	• citrate biodegradability • metal transport (Cu^{2+}, Zn^{2+}) • P-removal interference using FeCl_3 and $\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$ • general response of treatment system	

TABLE 2. TYPICAL EXPERIMENTAL DESIGN FOR CITRATE JAR TESTING

Jar #	Detergent Concentration as H_3Cit	Chemical Precipitant Concentration*		
		$FeCl_3$	$Al_2(SO_4)_3$	$Ca(OH)_2$
		as Fe^{3+}	as Al^{3+}	as $Ca(OH)_2$
1	0	0	0	0
2	0	5	5	50
3	0	10	10	100
4	0	20	20	200
5	0	30	30	300
6	10	5	5	50
7	10	10	10	100
8	10	20	20	200
9	10	30	30	300
10	20	5	5	50
11	20	10	10	100
12	20	20	20	200
13	20	30	30	300
14	30	5	5	50
15	30	10	10	100
16	30	20	20	200
17	30	30	30	300
18	0	0	0	0

* all values in mg/l.

Jar testing was also carried out to determine the effect of carbonate based detergent additions on phosphorus removal using hydrated lime. The initial alkalinity of the sewage was determined and appropriate quantities of the detergent (67% as $CaCO_3$) were added to give a 10%, 25% and 50% increase in the raw wastewater alkalinity. The jar testing procedure was the same as for the citrate detergent. The initial wastewater alkalinity varied from 190 to 325 mg/l as $CaCO_3$.

2.2 Bench Scale Studies - Activated Sludge System

To evaluate the influence of temperature on citrate detergent biodegradability and heavy metal (Cu^{2+} and Ni^{2+}) transport through a biological treatment process, three 10-litre continuous flow bench scale activated sludge reactors were operated in parallel. The reactor influent was raw, degrittied sewage from the Burlington Skyway Sewage Treatment Plant. For the various experimental conditions, the first reactor influent was supplemented with 12 mg/l detergent as citric acid, the second with 600 $\mu\text{g/l}$ Cu^{2+} and 600 $\mu\text{g/l}$ Ni^{2+} , and the third with 12 mg/l detergent as citric acid spiked with 600 $\mu\text{g/l}$ Cu^{2+} and 600 $\mu\text{g/l}$ Ni^{2+} . Only nickel and copper were considered in these studies since preliminary data gathered by Phizer Inc., (Foulds, 1973), indicated that only nickel and copper citrate complexes exhibited resistance to biodegradation. The metal concentrations evaluated were considered to be typical of a domestic wastewater with a heavy industrial contribution. Each reactor had a detention time of four hours and the mixed liquor suspended solids were maintained at 2,000 mg/l. The reactors were acclimatized for two weeks at 15°C, followed by a four-week test period during which citrate degradability and heavy metal transport were studied. The temperature was then dropped over the course of a week to 2°C, and testing as before continued for another four-week period. Analyses performed on the reactor influents and effluents included citric acid, BOD, TOC, total and filtered copper and total and filtered nickel.

2.3 Pilot Plant Studies - Carbonate Detergent Effect on Lime Phosphorus Removal System

An evaluation of the effect of carbonate detergent addition was carried out at the Wastewater Technology Centre using a 131 m³/d (20 gpm) chemical treatment pilot plant. A schematic of the pilot plant is shown in Figure 1. The treatment sequence included: (1) metering hydrated lime [Ca(OH)_2] with a volumetric dry feeder into a flash-mix tank where it was contacted with the raw, degrittied sewage for a period of five minutes, (2) flocculation by two flocculators in series for a total of 45 minutes, and (3) clarification in a settling

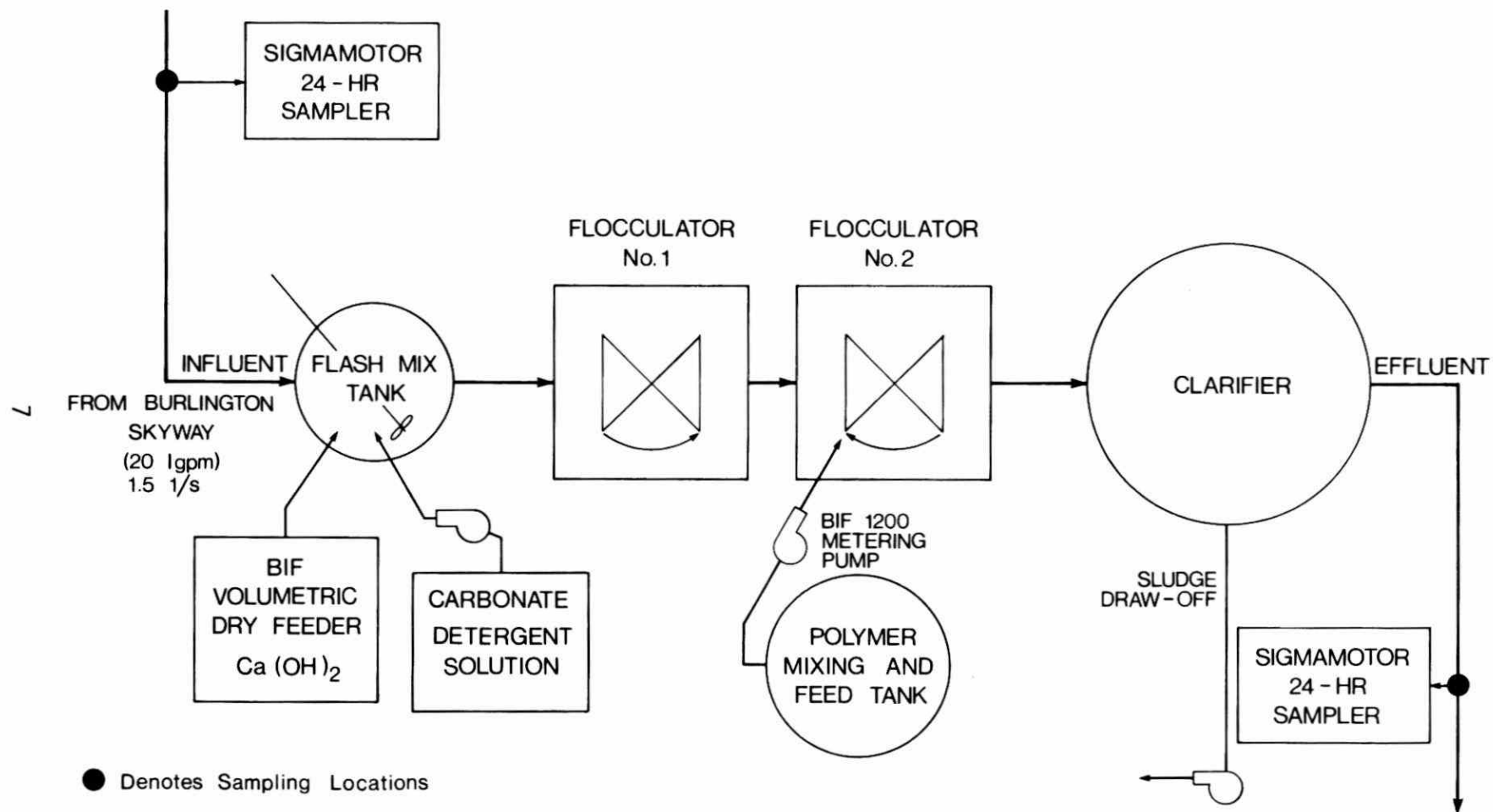


Fig. 1 SCHEMATIC DIAGRAM OF THE CHEMICAL PILOT PLANT, WITH LIME-DETERGENT ADDITIONS.

tank at a mean residence time of two hours and an overflow rate of $30 \text{ m}^3/\text{m}^2 \cdot \text{d}$ ($600 \text{ gpd}/\text{ft}^2$).

The experimental design for the pilot plant study is summarized in Table 3. Eight runs, each of five days duration, were conducted. The plant was operated at two lime dosages (240 mg/l to achieve pH 9.5 and 600 mg/l to achieve pH 11.5). For each of these conditions a commercially available carbonate detergent was added on the basis of increasing the initial wastewater alkalinity by 0%, 10%, 25% and 50%. The detergent was 71% Na_2CO_3 by weight (67% as CaCO_3) so the calculated detergent dosages required to increase the initial wastewater alkalinity ($\sim 240 \text{ mg/l}$ as CaCO_3) by 10%, 25% and 50% were 38, 95 and 190 mg/l detergent ($24, 60$ and 120 mg/l as CaCO_3). An extensive sampling and analytical program, summarized in Table 4, was conducted for each experimental condition. Sampling locations are indicated in Figure 1.

TABLE 3. EXPERIMENTAL PROGRAM FOR PILOT PLANT CARBONATE DETERGENT RUNS

Lime Dosage	Detergent Addition
Low Lime 240 mg/l as $\text{Ca}(\text{OH})_2$	0 - Baseline 10% Increase in baseline alkalinity 25% Increase in baseline alkalinity 50% Increase in baseline alkalinity
High Lime 600 mg/l as $\text{Ca}(\text{OH})_2$	0 - Baseline 10% Increase in baseline alkalinity 25% Increase in baseline alkalinity 50% Increase in baseline alkalinity

2.4 Full Scale Studies - Citrate Detergent

The experiments were designed to investigate the impact of citrate based detergent additions on a full scale, activated sludge, municipal wastewater treatment plant at Waterdown, Ontario. This plant, with a design capacity of $1360 \text{ m}^3/\text{d}$ ($300,000 \text{ gpd}$) is shown

schematically in Figure 2. Details of the Waterdown Wastewater Treatment Plant are described by Wei et al (1976). During this study, only one aeration tank was in operation since the plant influent flow ranged from 454 to 908 m³/d (100,000 to 200,000 gpd). The residence time in the aeration tank varied from 5.8 to 11.6 hours.

TABLE 4. SAMPLING LOCATIONS AND ANALYSES FOR PILOT PLANT CARBONATE DETERGENT EXPERIMENTS

Location	Frequency	Parameter
Influent and Effluent	Three 8-hour composite samples each day	BOD ₅ Total Phosphorus Soluble Phosphorus Total Organic Carbon Suspended Solids Volatile Suspended Solids pH Total Alkalinity Total Hardness Soluble Reactive Silica Sodium

A commercial product (20% citric acid by weight) imported from Sweden was used in the study. The experimental program examined citrate biodegradability, the effects of citrate on ferric chloride and alum phosphorus removal systems, and metal transport (Cu²⁺ and Zn²⁺) through the treatment plant. The six phases of the experimental program summarized in Table 5, extended from September, 1973 to March, 1974, with approximately four weeks for each phase.

Phase 1 was run to provide baseline reference data so that the results of the other study phases could be compared. Phases 2 and 3 were carried out to determine plant behaviour at two citrate levels bracketing conditions if all existing laundry detergents were replaced by a citrate based product. Earlier citrate substitution studies by Shannon and Kamp (1973) had shown that wastewater citrate levels

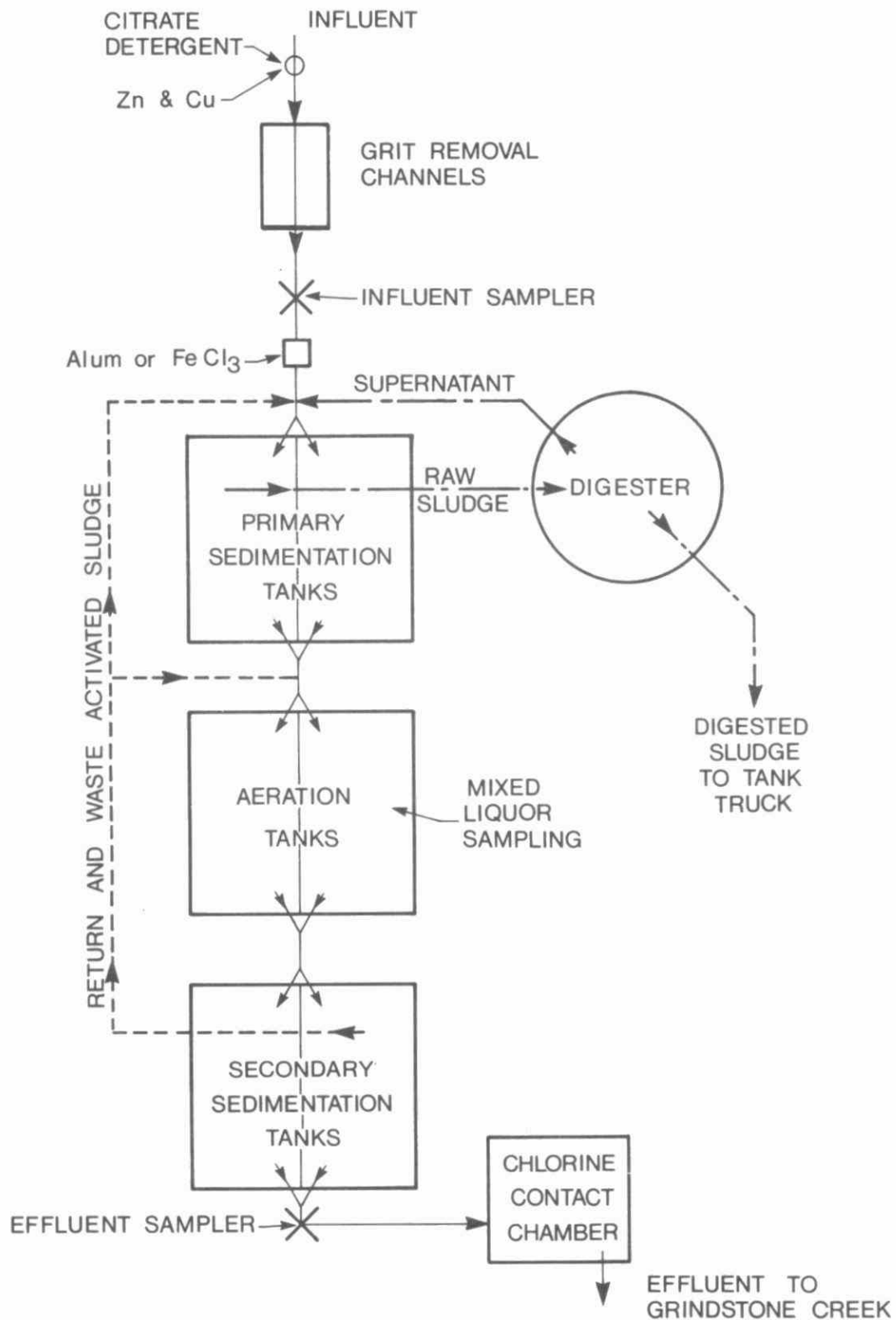


Fig.2 SCHEMATIC OF WATERDOWN WASTEWATER TREATMENT PLANT.

averaged 7.0 mg/l as citric acid when citrate based detergents were the only detergents in use. Phase 4 was designed to investigate metal transport at heavy metal loadings representative of a combined industrial-domestic wastewater. Phases 5 and 6 studied the effects of citrate on ferric chloride and alum phosphorus removal systems. The alum and ferric chloride dosages required to meet an effluent total phosphorus level of 1 mg/l P or less were established in earlier work by Wei et al (1976).

TABLE 5. EXPERIMENTAL PROGRAM FOR FULL SCALE CITRATE DETERGENT RUNS

Phase	Detergent Addition as H ₃ Cit* (mg/l)	Total Metal Level (µg/l)	Phosphorus Removal System
1	None	Baseline	None
2	6	Baseline	None
3	12	Baseline	None
4	6	200 - Copper 400 - Zinc	None
5	6	200 - Copper 400 - Zinc	Alum (5 mg/l as Al ³⁺) to the primary
6	6	200 - Copper 400 - Zinc	Ferric Chloride (10 mg/l as Fe ³⁺) to the primary

* Citric Acid

Detergent and metal solution storage tanks and feed facilities were located at a manhole approximately 30 m (100 feet) upstream of the plant influent works. The alum and ferric chloride solutions were metered into the raw sewage immediately prior to the primary settling tanks. Samples were collected on a routine basis five days per week for the duration of the study. The sampling points and chemical feed locations are indicated in Figure 2. The chemical parameters, sampling locations and monitoring frequency are presented in Table 6.

TABLE 6. CHEMICAL PARAMETERS MONITORED DURING FULL SCALE EXPERIMENTS

Parameter	Sampling Procedure	Location	Frequency
Citrate	Grab	Influent and Effluent	Two per day, diurnal hourly sampling once every five days
Aluminum Iron	Composite	Influent and Effluent	One flow proportioned composite per day
Copper Zinc	Grab	Mixed Liquor	Composite of two grab samples per day
BOD ₅ Total Organic Carbon Suspended Solids	Composite	Influent and Effluent	One flow proportioned composite sample per day
Hardness	Grab	Influent and Effluent	Two per day
MLSS SVI	Grab	Aeration Tank	Two per day
Temperature	In situ	Influent, Aeration Tank, and Effluent	Two per day
Total Phosphorus	Composite	Influent and Effluent	One flow proportioned composite per day

2.5 Wastewater Treatment Plant Survey

To obtain estimates of citrate baseline levels in raw and treated domestic wastewater, a random sampling of the influent and effluent of 32 southern Ontario wastewater treatment plants was carried out. The grab samples were collected, preserved and returned to the Burlington laboratory for analyses.

2.6 Analytical Procedures

The majority of the analyses performed during this project were conducted according to Standard Methods (APHA et al, 1971). The exceptions are described in the following:

1. The samples collected for citrate analyses were

- filtered immediately through a 0.45 μ membrane filter and preserved with concentrated hydrochloric acid at pH 2. The analyses were performed according to a fluorometric procedure described by Afghan et al (1974); and
2. The aluminum, iron, copper, nickel and zinc analyses were performed according to Traversy (1971).

3 RESULTS AND DISCUSSION

3.1 Effect of Citrate Detergent on Chemical Phosphorus Removal - Jar Tests

Typical effects of citrate detergent additions (expressed in mg/l as H_3Cit) on ferric chloride, alum and lime phosphorus removal jar test results using degrittied raw sewage are shown in Figures 3, 4, and 5, respectively. A definite trend toward reduced phosphorus removal was observed as the citrate level increased above 10 mg/l.

Higher soluble iron/aluminum levels at increased citrate detergent addition levels and a similar effect in tests using pure sodium citrate additions showed that this interference with phosphorus removal was due to complexing of the precipitant cation (Fe^{3+} , Al^{3+} , Ca^{2+}) by the citrate, and not due to the surfactant interfering with the floc settling properties.

As pointed out earlier, citrate substitution studies by Shannon and Kamp (1973) showed that wastewater citrate levels averaged 7 mg/l (as H_3Cit) when citrate based detergents were the only detergents in use. This is in the lower range of the levels investigated here. However, the jar test data suggests that if all detergents now in use were to be replaced by citrate based detergents (20% H_3Cit by weight), the ferric chloride, alum, and lime requirements to achieve a given level of phosphorus removal (i.e., 1 mg/l residual P) in primary treatment systems may be expected to increase.

3.2 Effect of Carbonate Based Detergent on Phosphorus Removal with Lime - Jar Tests

The effect of the addition of a carbonate based detergent [71% Na_2CO_3 by weight (67% as $CaCO_3$)] on a lime phosphorus removal system is shown in Figure 6. Although there was a slight tendency toward reduced phosphorus removals as the carbonate addition increased, this was not as pronounced as in the tests with citrate detergent.

Shannon and Kamp (1973) showed that complete substitution of carbonate detergents for existing detergents increased the baseline alkalinity by approximately 70 mg/l (18%). The data presented in

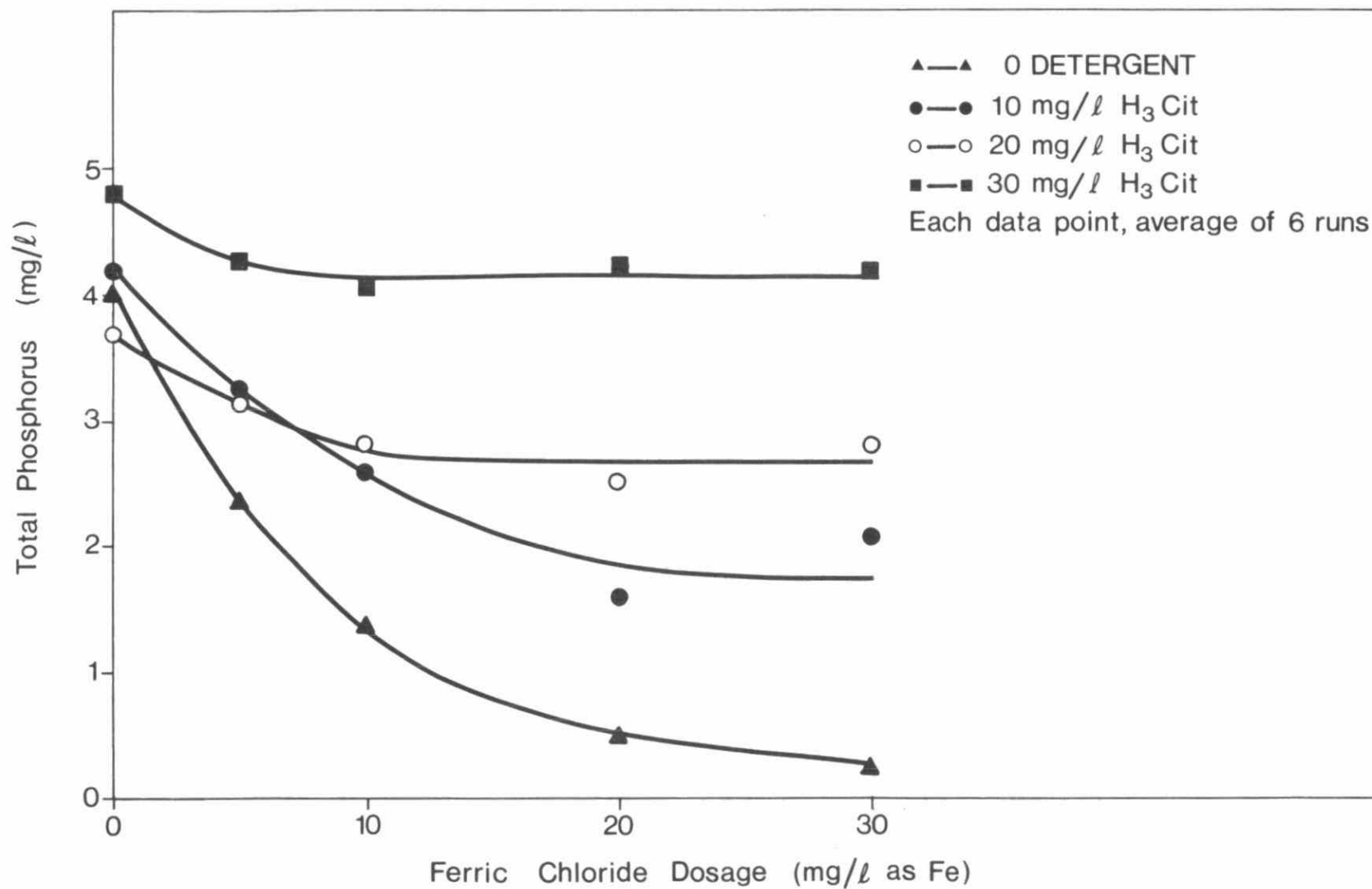


Fig.3 EFFECT OF CITRATE BASED DETERGENT ON PHOSPHORUS REMOVAL BY FERRIC CHLORIDE. (JAR TESTS)

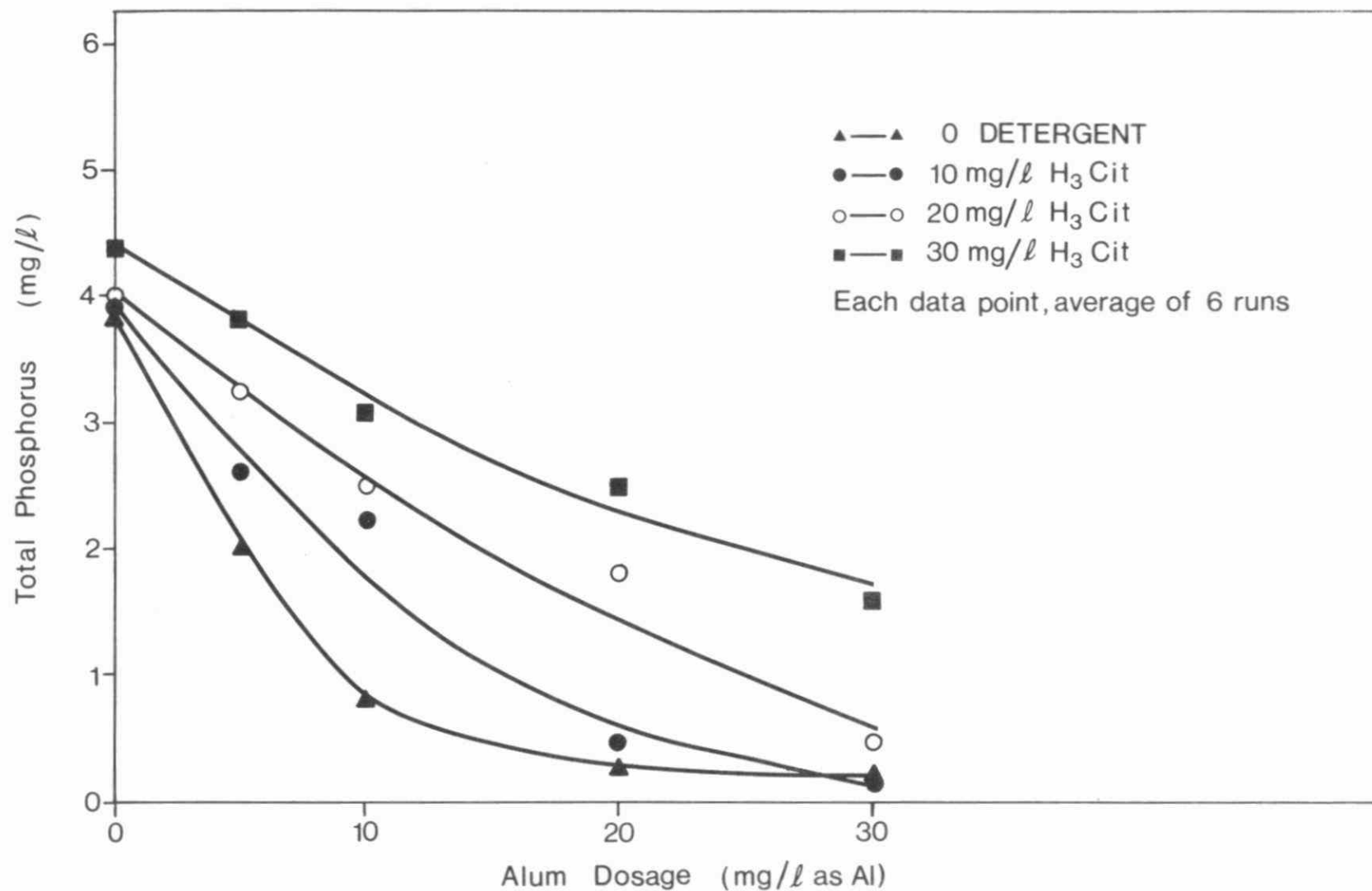


Fig.4 EFFECT OF CITRATE BASED DETERGENT ON PHOSPHORUS REMOVAL BY ALUM (JAR TESTS)

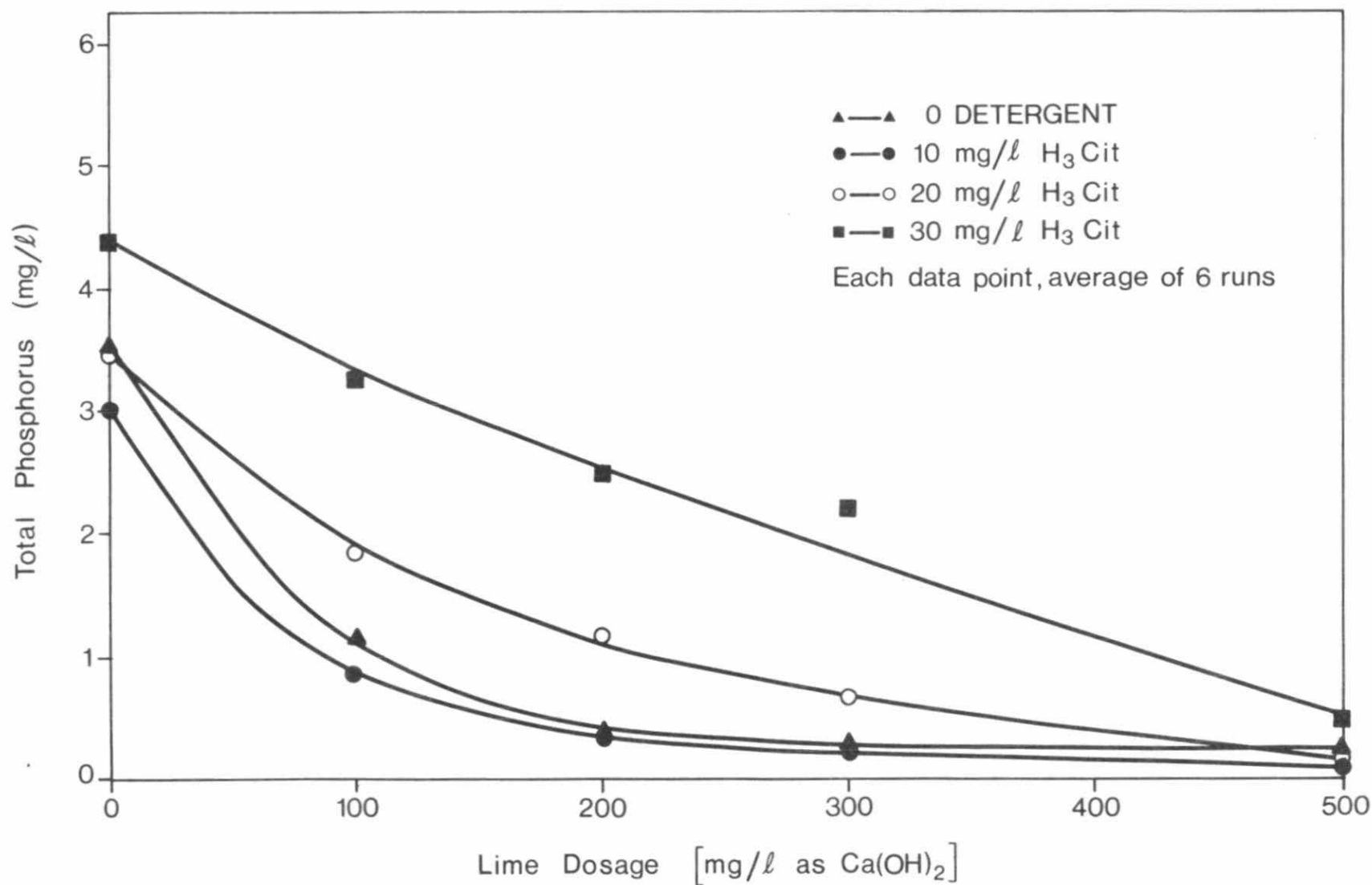


Fig.5 EFFECT OF CITRATE BASED DETERGENT ON PHOSPHORUS REMOVAL BY LIME. (JAR TESTS)

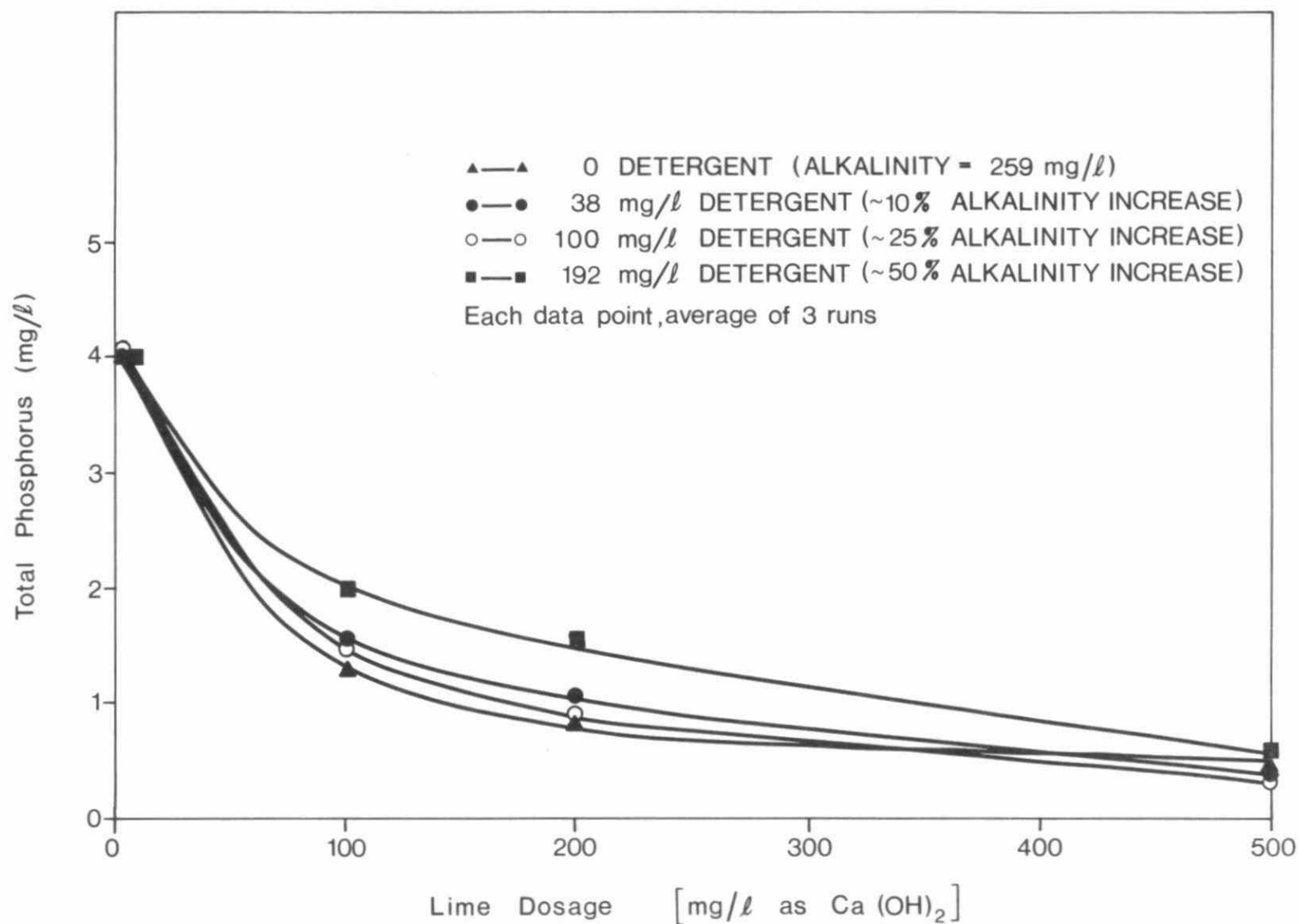


Fig.6 EFFECT OF CARBONATE BASED DETERGENT ON PHOSPHORUS REMOVAL BY LIME. (JAR TESTS)

Figure 6 shows no discernible difference in phosphorus removal efficiency between the baseline (no detergent) and 25% alkalinity increase levels. Therefore, the impact of complete utilization of carbonate based detergents on lime phosphorus removal systems may be expected to be insignificant.

3.3 Bench Scale Activated Sludge Degradability and Metal Transport Experiments with Citrate Based Detergent

The results obtained during the bench scale activated sludge experiments with citrate detergent are summarized in Table 7. Citrate degradation was only slightly affected by lowering the wastewater temperature from 15°C to 2°C. For the control reactor (R1), the percent removal was reduced from 96% to 93%. For the nickel/copper reactor (R2) degradation decreased from 81% to 78%. This slight decrease in citrate degradability with temperature was also observed in the full scale studies. The presence of higher effluent citrate levels in the reactor being fed copper, nickel, and citrate (R3) indicated that some of the citrate was being tied up in a copper or nickel complex which was not as biodegradable as the citrate in the control reactor. However, there was no evidence that the presence of the citrate detergent increased copper or nickel transport through the reactor.

3.4 Pilot Plant Experiments on the Carbonate Detergent

Physical-chemical pilot plant experiments [131 m³/d (20 gpm)] were carried out to determine the effects of carbonate detergent additions upon: (1) a low lime [240 mg/l as Ca(OH)₂], and (2) a high lime [600 mg/l as Ca(OH)₂] phosphorus removal system. The experimental design for this study was discussed in Section 2.3. Results for the low and high lime systems are summarized in Tables 8 and 9, respectively. The low lime system shows a definite trend of increasing total and filtered effluent phosphorus with increasing carbonate detergent additions. The high lime phosphorus removal performance did not appear to be significantly affected at any of the carbonate addition

TABLE 7. SUMMARY OF BENCH SCALE ACTIVATED SLUDGE EXPERIMENTS ON CITRATE DETERGENT

Parameter	T°C	Reactor Conditions (See Section 2.2)					
		R1 Wastewater Plus Citrate Feed		R2 Wastewater Plus Copper and Nickel		R3 Wastewater Plus Citrate Plus Copper and Nickel	
		Inf.	Eff.	Inf.	Eff.	Inf.	Eff.
Citrate (mg/l as H ₃ Cit)	2°C	13.8	0.9	0.5	0.5	15.8	3.5
	15°C	11.4	0.4	0.4	0.2	13.3	2.5
Copper (µg/l)	2°C	86 ¹ 71 ²	67 63	647 555	623 515	675 563	569 523
	15°C	66 50	36 30	625 504	500 352	557 447	401 277
Nickel (µg/l)	2°C	9 9	8 8	623 567	597 515	581 521	511 480
	15°C	13 11	12 11	658 620	580 512	647 623	597 540
BOD ₅ (mg/l)	2°C	72	36	56	36	68	36
	15°C	69	21	50	20	75	24
TOC (mg/l)	2°C	31	15	21	12	32	17
	15°C	27	9	16	8	28	11

¹ Total metal concentration.² Filtered (0.45 µ) metal concentrations.

TABLE 8. EFFECTS OF CARBONATE DETERGENT ADDITIONS ON A LOW LIME
(≈ 240 mg/l) PHOSPHORUS REMOVAL SYSTEM

Parameter	Detergent Addition Level - % Increase in Total Alkalinity							
	0		10		25		50	
	Inf.	Eff.	Inf.	Eff.	Inf.	Eff.	Inf.	Eff.
Total P (mg/l)	3.4	1.2	3.1	1.4	3.3	1.8	4.3	2.4
Filtered P (mg/l)	2.6	0.9	2.5	0.9	2.5	1.1	2.9	1.5
Hardness (mg/l as CaCO_3)	306	226	298	203	292	189	274	181
Alkalinity (mg/l as CaCO_3)	241	164	241	171	237	193	233	244
Dissolved Silica (mg/l)	2.9	2.2	3.6	3.1	2.7	3.2	2.0	4.5
Sodium (mg/l)	69	68	76	81	80	100	76	120

TABLE 9. EFFECTS OF CARBONATE DETERGENT ADDITIONS ON A HIGH LIME
(≈ 600 mg/l) PHOSPHORUS REMOVAL SYSTEM

Parameter	Detergent Addition Level - % Increase in Total Alkalinity							
	0		10		25		50	
	Inf.	Eff.	Inf.	Eff.	Inf.	Eff.	Inf.	Eff.
Total P (mg/l)	4.2	0.3	4.7	0.2	5.1	0.5	5.3	0.4
Filtered P (mg/l)	2.3	0.1	2.5	0.1	2.5	0.1	2.1	0.2
Hardness (mg/l as CaCO_3)	314	348	301	373	281	288	258	239
Alkalinity (mg/l as CaCO_3)	238	292	236	342	239	307	234	330
Dissolved Silica (mg/l)	4.7	3.8	3.5	3.7	3.7	4.4	2.6	3.2
Sodium (mg/l)	99	79	92	100	105	130	85	121

levels. At least 90% removal was achieved in all cases. Thus, full scale usage of carbonate detergents which increase the raw wastewater alkalinity by 10% to 20% should have no significant effect on lime phosphorus removal systems. This confirms the results of the jar testing program discussed earlier. (Note: Wastewaters with a lower baseline alkalinity may experience greater percentage alkalinity increases for a given input of carbonate detergent.) A survey of 20 Ontario municipal wastewaters by Prested et al (1976), found that alkalinities varied from 110 to 425 mg/l as CaCO_3 with an average of 240 mg/l as CaCO_3 . Consequently, the majority of wastewaters would probably respond to carbonate detergents in a manner similar to the wastewater used here.

Other effects of the carbonate detergent addition are noted as follows:

1. The low lime system effluent alkalinity increased in proportion to detergent added; and
2. effluent sodium and dissolved silica increased for both low and high lime systems roughly in proportion to detergent added. This is attributable to the sodium and silica content of the detergent. At practical addition levels, the magnitude of sodium and dissolved silica increase would be in the order of 20% to 30%, respectively. Shannon and Kamp (1973) noted a similar effect in their full scale detergent substitution studies.

3.5 Full Scale Citrate Addition Study at Waterdown

A complete summary of results from this phase of the study is presented in the Appendix. These results are discussed below under the appropriate headings.

3.5.1 Citrate removals

A summary of the citrate degradation data is given in Table 10. Based on a "t" - test, effluent citrate levels during Phases 2,

3, 4, and 5 were not statistically different. As anticipated, the removal of citrate through the plant was high - 90% average removal over a six-month period which included fall and winter operation. These removals were slightly lower than the up to 96% removals observed in the bench scale studies (Section 3.3). Citrate degradation was significantly influenced at wastewater temperatures less than 11°C (Figure 7). Similar observations for NTA degradation in activated sludge, but to a much larger degree, were reported on by Eden et al (1972).

TABLE 10. CITRATE DEGRADATION DURING FULL SCALE PROGRAM

Phase	Conditions	Citrate (mg/l as H ₃ Cit)			Wastewater Temp. (°C)
		Inf.	Eff.	% Removal	
1	Baseline	2.9	0.81	72.8	19.6
2	6 mg/l H ₃ Cit	6.4	0.40	93.8	19.4
3	12 mg/l H ₃ Cit	11.5	0.33	97.1	16.8
4	6 mg/l H ₃ Cit 200 µg/l Copper 400 µg/l Zinc	5.4	0.27	95.1	14.5
5	6 mg/l H ₃ Cit 200 µg/l Copper 400 µg/l Zinc Alum addition at 5 mg/l as Al ³⁺	6.4	0.51	92.0	9.8
6	6 mg/l H ₃ Cit 200 µg/l Copper 400 µg/l Zinc Ferric Chloride addition at 10 mg/l as Fe ³⁺	6.7	0.92	86.3	8.8

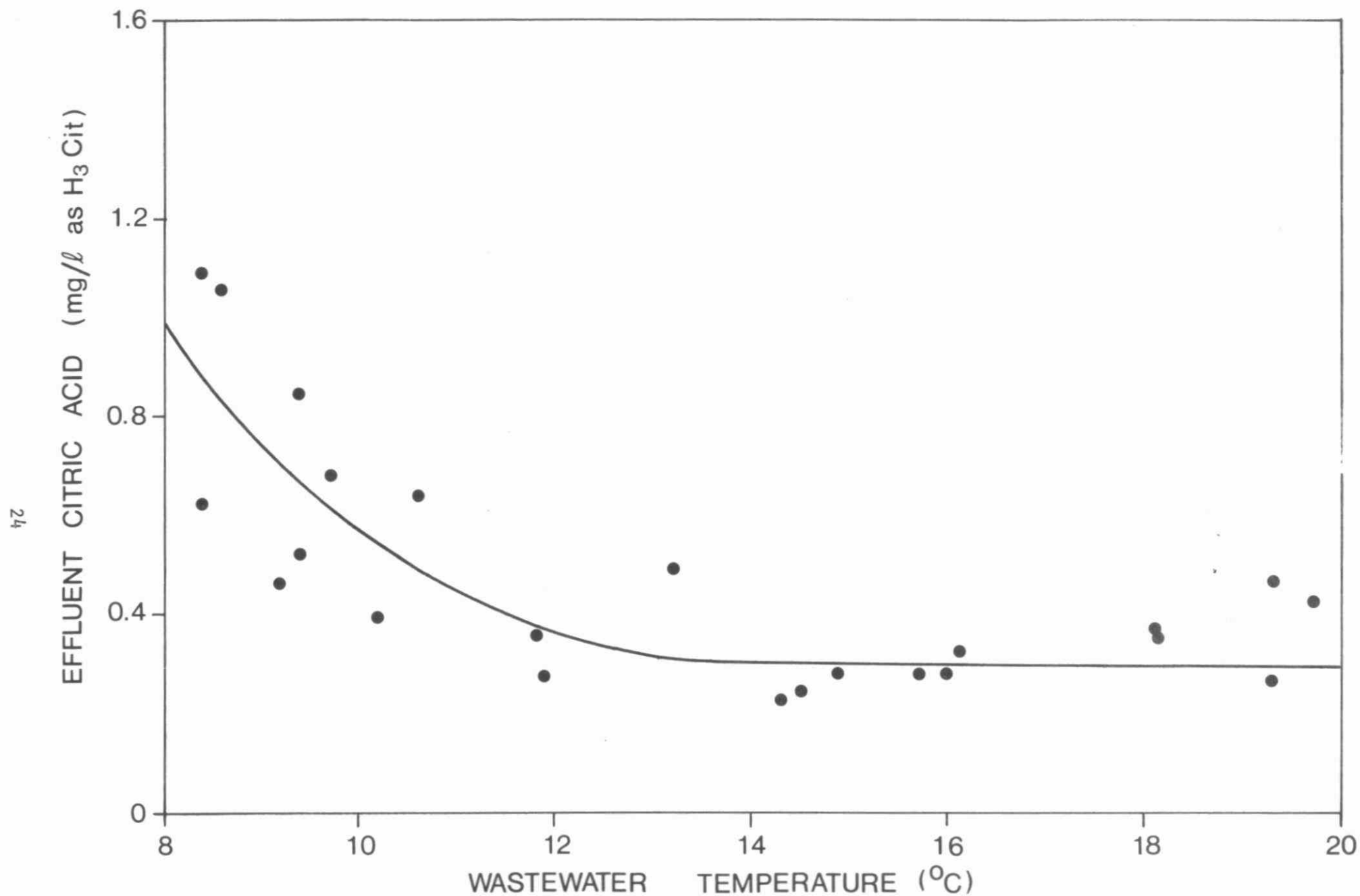


Fig.7 RELATIONSHIP BETWEEN EFFLUENT CITRIC ACID LEVELS FROM AN ACTIVATED SLUDGE PLANT AND WASTEWATER TEMPERATURE.

3.5.2 BOD₅ and TOC effects

Average values for BOD₅ and TOC in the plant influent and effluent for each phase and the calculated contribution due to citric acid are shown in Table 11. The carbon contribution due to citric acid was calculated using laboratory measured values of 0.55 mg BOD₅/mg of H₃Cit and 0.35 mg of TOC/mg of H₃Cit and the measured citric acid concentrations for each phase. Based on these data, full scale citrate detergent usage would lead to small increases in wastewater BOD₅ and TOC (influent BOD₅ and TOC increases of 3% and 5%, respectively). Effluent BOD₅ and TOC increases would be lower (in the order of 1% to 2%). It is thus evident that additional oxygen demands due to citrate at full detergent substitution levels are insignificant.

TABLE 11. BOD₅ AND TOC DATA DURING FULL SCALE CITRATE PROGRAM

Phase (see Table 10 for Conditions)	Influent				Effluent			
	BOD ₅ mg/l	% due to H ₃ Cit*	TOC mg/l	% due to H ₃ Cit*	BOD ₅ mg/l	% due to H ₃ Cit*	TOC mg/l	% due to H ₃ Cit*
1	134	1.2	52.1	2.0	8	5.4	14.7	1.9
2	140	2.5	44.0	5.0	12	1.9	13.1	1.0
3	168	3.8	75.2	5.3	11	1.6	13.3	0.9
4	102	2.9	44.6	4.2	14	1.1	13.1	0.7
5	94	3.8	47.0	4.7	8	3.4	8.5	2.1
6	139	2.7	50.3	4.6	15	3.5	12.4	2.6

* Calculated, based on quantity of citrate determined.

3.5.3 Citrate and metals

The baseline copper and zinc levels were typical of a domestic wastewater, whereas the augmented conditions were typical of a combined industrial-domestic wastewater. Copper and zinc data are summarized in Table 12. Effluent copper and zinc levels were not significantly different over the first three phases, indicating no increased transport of these metals by citrate detergent addition. During the final three phases, where copper and zinc were added, the effluent copper concentrations

TABLE 12. COPPER AND ZINC DATA DURING FULL SCALE CITRATE PROGRAM

Phase (see Table 10 for Conditions)	Copper ($\mu\text{g/l}$)				Zinc ($\mu\text{g/l}$)			
	Inf.	Eff.	M.L.*	% Rem.	Inf.	Eff.	M.L.	% Rem.
1	102	29	1710	71.6	136	36	2260	73.5
2	114	22	1510	80.7	186	56	2060	69.9
3	116	23	1350	80.2	159	58	1830	63.5
4	198	46	3310	76.8	520	54	3300	89.6
5	160	32	2580	80.0	388	44	3430	88.6
6	192	34	2730	82.3	318	35	3620	89.0

* Copper and zinc concentrations in aeration tank mixed liquor.

were correspondingly higher, but no increase in effluent zinc was observed. For both metals the mixed liquor metal concentrations were increased roughly proportional to the influent. The alum and ferric chloride added for phosphorus removal reduced the effluent copper and zinc concentrations. No correlation was obtained between metal and citric acid in the effluent. Under these experimental conditions, there was no evidence of increased heavy metal (Cu^{2+} and Zn^{2+}) transport due to the addition of citric acid based detergent.

3.5.4 Citrate and phosphorus removal

In a previous jar testing and full scale study (Wei et al, 1976) at the Waterdown Wastewater Treatment Plant, it was determined that in order to achieve an average effluent total phosphorus level of 1 mg/l, primary additions of ferric chloride at 10 mg/l as Fe^{3+} or of alum at 5 mg/l as Al^{3+} , were required. For the present study, aluminum and ferric chloride were added at these levels to determine if effluent phosphorus concentrations would be affected by the citrate additions. A summary of results is presented in the Appendix. The average effluent total phosphorus during the alum addition period of this study was 0.7 mg/l, whereas, during the ferric chloride addition period, it averaged 1.12 mg/l. For the alum addition period, the

addition of citrate at 6 mg/l as H₃Cit did not interfere with phosphorus removal. During the ferric chloride period, the effluent total phosphorus was slightly higher than the expected 1 mg/l; however, the effect was minimal.

It is unlikely that a complete switch to citrate detergents would increase average wastewater citrate levels to greater than 10 mg/l as H₃Cit (Shannon and Kamp, 1973); thus the impact on phosphorus removal systems would be negligible.

It is important to note that, while jar test data (Section 3.1) indicated that citrate addition may interfere with phosphorus removal in primary plants, the full scale showed no interference with the activated sludge process. This is likely due to the fact that citrate was about 90% removed by biological treatment.

3.5.5 Citrate and hardness

Influent and effluent hardness was measured during the six experimental phases and are summarized in Table 13. The influent hardness during the first three phases is relatively constant, but the corresponding effluent values increased slightly with increasing citrate detergent additions. The influent and effluent hardness values for Phases 4 to 6 are higher than those for Phases 1 to 3. This suggests that the effluent citric acid may be associated with calcium and/or magnesium rather than with the metals. Calcium plus magnesium was in an approximately 10^3 to 10^4 factor molar excess over copper or zinc in the effluent and this may explain why there was no relation between these metals and citric acid in the effluent.

TABLE 13. HARDNESS DATA DURING FULL SCALE CITRATE PROGRAM

Phase (see Table 10 for Conditions)	Influent (mg/l CaCO ₃)	Effluent (mg/l CaCO ₃)
1	298	276
2	287	285
3	314	303
4	359	349
5	359	353
6	375	361

3.6 Survey of Baseline Citrate Levels

A summary of baseline citrate concentrations determined at 32 Ontario wastewater treatment plants is presented in Table 14.

Influent and effluent grab samples were taken over a three-day period during which the average ambient temperature varied from a high of 5°C to a low of 0°C. As shown in Table 14, a second grab sample was taken at some plants on a later date. In the data analyses, the two high citrate influent concentrations for the cities of Chatham and Preston were not included. This was because they were considered to be an anomaly with respect to all other data collected and this is attributed to the intensive food processing operations in these cities.

As shown in Figure 8, the median baseline influent citrate concentration approximates 1.1 mg/l. Primary treatment reduced this to a median effluent concentration of approximately 0.4 mg/l, while activated sludge treatment brought about a reduction to 0.2 mg/l citrate. This corresponds to 55% and 92% citrate reduction due to the respective waste treatment processes.

TABLE 14. CITRATE SURVEY OF ONTARIO WASTEWATER TREATMENT PLANTS

Location	Plant Type	Citrate (mg/l as H ₃ Cit)		Location	Plant Type	Citrate (mg/l as H ₃ Cit)	
		Influent	Effluent			Influent	Effluent
Burlington (Skyway)	Act. Sludge	1.1	<0.1	C.F.B. Borden	Primary	1.2	0.4
Burlington	Act. Sludge	0.4	<0.1	Dundas	Primary	1.4	0.1
Burlington (Eliz.G)	Act. Sludge	0.6	<0.1	Fort Erie	Primary	0.8	1.2
Chatham	Act. Sludge	2.9 (11.3)*	0.3 (0.4)*	Niagara Falls	Primary	1.0	0.5
Chippawa	Act. Sludge	1.2	0.1			1.1	1.1
Dunville	Act. Sludge	1.6	1.1			1.4	<0.1
		1.6	0.5	Port Colborne	Primary	0.8	0.3
Elmira	Act. Sludge	1.9	1.1			1.2	0.2
		2.3	1.1	Port Dalhousie	Primary	1.4	1.4
Galt	Act. Sludge	1.1	0.2			2.1	0.9
Guelph	Act. Sludge	0.8	0.1	Port Dover	Primary	0.6	0.5
		0.7	0.1			0.4	0.1
Hamilton	Act. Sludge	0.7	<0.1	Welland	Primary	0.4	0.1
Hespeler	Act. Sludge	0.7	<0.1	Grimsby	Lagoon	1.0	<0.1
Kitchener	Act. Sludge	1.5	0.2			1.1	0.2
		1.2	0.4	Petersburg	Lagoon	0.9	0.1
London (Adelaide)	Act. Sludge	1.2	0.1	* Data anomaly attributed to food processing industry and not included in Figure 8.			
London (Greenway)	Act. Sludge	1.1	<0.1				
London (Oxford)	Act. Sludge	1.6	0.5				
London (Vauxhall)	Act. Sludge	0.4	0.1				
Oakville (SW)	Act. Sludge	0.7	0.1				
Oakville (SE)	Act. Sludge	0.8	0.4				
Peterborough	Act. Sludge	0.9	0.3				
Port Weller	Act. Sludge	0.9	<0.1				
Preston	Act. Sludge	1.8 (27.6)*	0.9 (0.4)*				
Waterloo	Act. Sludge	1.3	0.2				
		1.2	0.2				

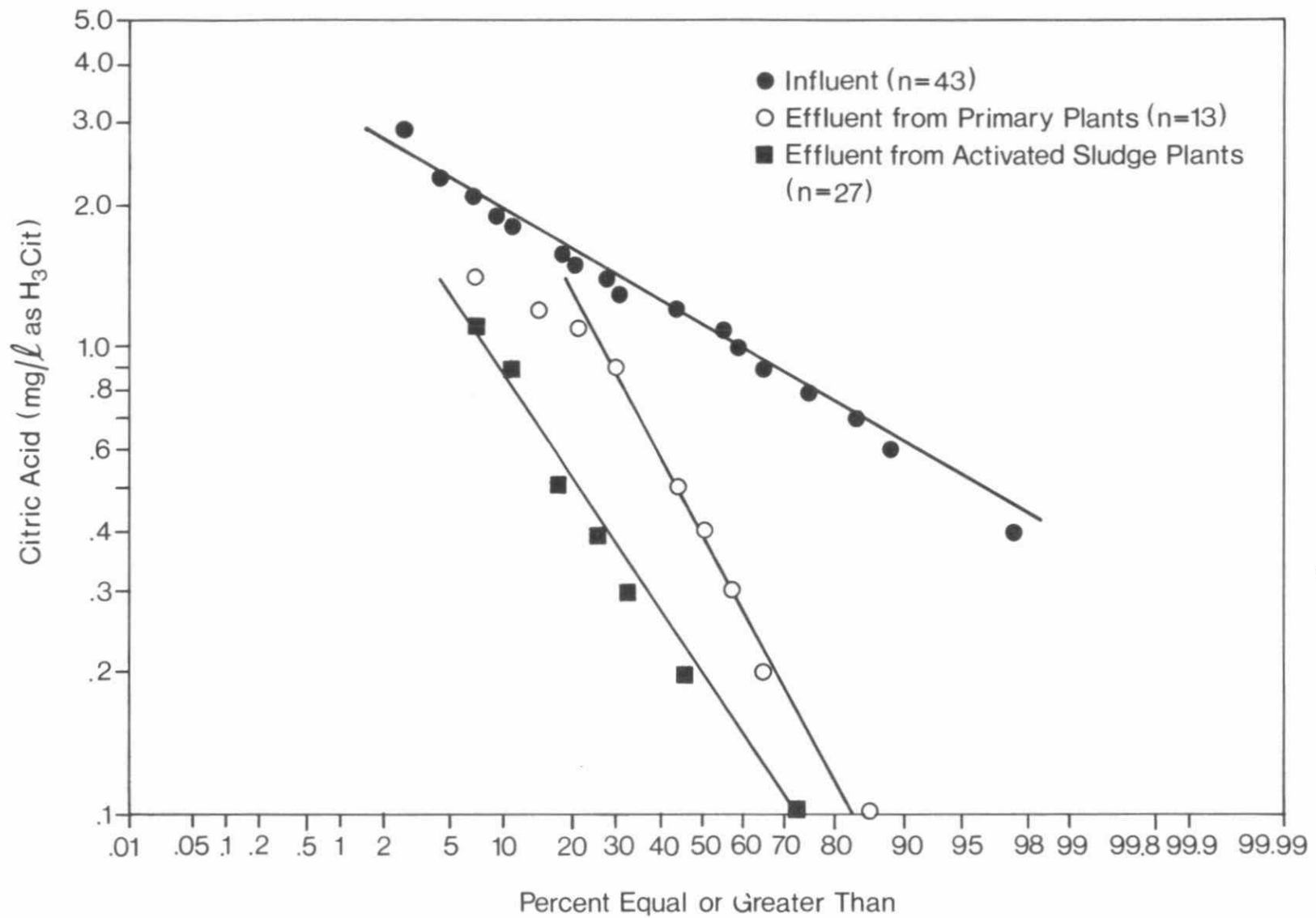


Fig.8 INFLUENT-EFFLUENT CITRIC ACID CONCENTRATIONS AT ONTARIO WASTEWATER TREATMENT PLANTS.

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APPENDIX

Full Scale Citrate Degradation Program Data Summary

PHASE		Baseline		H ₃ Cit. - 6 mg/l		H ₃ Cit. - 12 mg/l		H ₃ Cit. - 6 mg/l Cu - 200 ug/l Zn - 400 ug/l		H ₃ Cit. - 6 mg/l Cu - 200 mg/l Zn - 400 mg/l Al - 5 mg/l		H ₃ Cit. - 6 mg/l Cu - 200 ug/l Zn - 400 ug/l Fe - 10 mg/l	
PARAMETER		1 influent effluent	Mixed Liquor	2 influent effluent	Mixed Liquor	3 influent effluent	Mixed Liquor	4 influent effluent	Mixed Liquor	5 influent effluent	Mixed Liquor	6 influent effluent	Mixed Liquor
CITRATE mg/l		2.9 0.81		6.4 0.40		11.5 0.33		5.4 0.27		6.4 0.51		6.7 0.92	
ORGANICS	BOD ₅ (Total)mg/l	134 8		140 12		168 11		102 14		94 8		139 15	
	TOC mg/l	52 15		44 13		75 13		45 13		47 9		50 12	
HEAVY METALS (TOTAL)	Cu ug/l	102 29	1710	114 22	1510	116 23	1350	198 46	3310	160 32	2580	192 34	2730
	Zn ug/l	136 36	2260	186 56	2060	159 58	1830	520 54	3300	388 44	3430	318 35	3620
	Al ug/l	160 30	6070	220 50	6380	260 60	5960	400 70	9270	410 160	40700	190 60	10900
	Fe ug/l	540 240	17300	660 250	15800	680 160	13800	640 130	25700	920 120	21100	530 690	96600
PLANT OPERATION	MLSS mg/l		2370		2070		1980		2495		3100		2415
	SVI ml/g		160		177		126		100		29		40
	Temp. °C		19.6		19.4		16.8		14.5		9.8		8.8
PHOSPHORUS REMOVAL	Hardness mg/l as CaCO ₃	298 276		287 285		314 303		359 349		359 353		375 361	
	TP mg/l	5.6 5.1		4.8 4.2		5.4 4.0		3.2 3.2		3.5 0.7		4.0 1.2	
	FP mg/l	4.0 4.0		3.4 4.0		3.9 3.7		2.5 2.6		3.0 0.5		2.9 0.7	
	SS mg/l	132 6		195 7		138 20		83 18		120 16		122 22	

TABLE A-1. FULL SCALE - CITRATE DEGRADATION PROGRAM - DATA SUMMARY

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1977

Effect of citrate and carbonate
based detergents on wastewater
characteristics and treatment /
Shannon, E. E.
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